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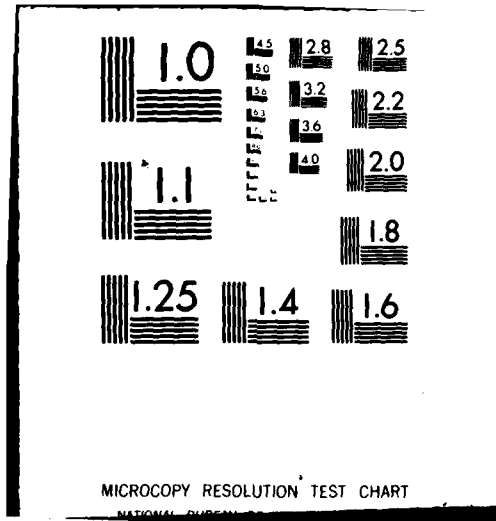
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ROYAL AIRCRAFT ESTABLISHMENT

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Technical Report 81145

November 1981

**THE DESIGN AND USE OF
A VISCOMETER FOR THE STUDY
OF REACTING RESIN SYSTEMS**

by

R. Childs

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Procurement Executive, Ministry of Defence
Farnborough, Hants

R O Y A L A I R C R A F T E S T A B L I S H M E N T

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Received for printing 26 November 1981

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SUMMARY

This Report describes a viscometry system which has been developed specifically to study reacting resin systems under predetermined temperature cycles such as those representative of the cure conditions used for carbon fibre reinforced epoxy resin laminates. The constraints placed upon the design both by the need to reduce the number of variables and by the nature of the material under test are discussed together with the methods adopted to overcome them. By use of an example, the viscometry system described is shown to provide a detailed understanding of what, at first sight, appears to be a simple process but is in fact unexpectedly complex.

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1 INTRODUCTION

The majority of the resin systems used in composite materials for aerospace applications require an elevated temperature cure. In addition most of the fabrication procedures require the application of both temperature and pressure to produce a satisfactory product. In this Report the processes discussed will primarily be those appropriate to fibre reinforced epoxy matrix materials fabricated in an autoclave. However, it should be noted that the system described is applicable to a much wider range of curing procedures.

For a variety of reasons - thermal inertia, power limitations, etc - the attainment of a given temperature requires a finite time. On the other hand, the pressure can be applied at any time during the cure cycle, its rate of application can be varied widely, and its magnitude is limited only by the strength of the vessel being used. However, very little experience is required to show that neither heat nor pressure can be applied in a haphazard manner; on the contrary there is only a limited range of conditions which will produce a satisfactory composite^{1,2} giving what might be called a process bandwidth, and it is apparent that the magnitude of this bandwidth will define the tolerance of the resin system to variations, or perturbations, in the cure procedures. If the bandwidth were zero then it would be impossible to develop a cure procedure, but in general such a bandwidth does exist and there can be derived empirically a sequence of actions necessary to produce the requisite composite quality taking into account the restraints enforced by the resin, by the size and nature of the product and its associated tooling. This whole procedure is known loosely as the cure cycle. During this cure cycle the elevated temperature will cause the resin to flow. Now the resin content of the composite before processing can be equal to, or greater than, that of the final product. In the former case, whilst resin flow might be desirable to give a homogeneous product, no loss can be tolerated; in the latter case however resin flow is necessary, because a known and finite loss must be permitted but not exceeded. The fact that resin flow takes place implies a certain level of viscosity and it seems reasonable to suppose that a knowledge of the viscosity characteristics of the resin system during a cure cycle might enhance the understanding of the nature of the conditions creating that cycle.

This Report describes a simple viscometer designed specifically to study reacting resin systems under conditions representative of autoclave cure cycles, although it is sufficiently versatile to deal with other methods of treatment. It has been called for obvious reasons the Resin Cure Cycle Simulator.

2 DESIGN AND CONSTRUCTION OF THE SIMULATOR

2.1 General description

Any viscometer can be considered as a combination of two components, the viscometer module and the apparatus by which its temperature of operation is governed. Normally this latter component is a constant temperature bath but since, in this case, a pre-determined temperature profile is required the apparatus is more complex. Also, because of the nature of the material being studied, the viscometer itself needs to be small and

expendable; two constraints which need not normally be considered. There are in addition a number of other components such as power supplies and data recorders which can conveniently be grouped under the general heading of auxiliaries. Thus the Simulator can be discussed in three parts:

- (i) the viscometer
- (ii) the heating system
- (iii) the auxiliaries.

The complete instrument is shown in Fig 1. On the right, in a separate rack, are two recorders, one to record the sample temperature and the other the viscosity. On the left is a frame supporting on the top the viscometer module comprising the heater bath, the resin container, the drive motor and the circulating pump. Flanking it on the left is the drive motor control and on the right the function generator which includes both the heating control and the circuits to generate the required temperature profile. Underneath are the power units for the heater and for the circulating pump.

2.2 Design of the viscometer module

2.2.1 General considerations

There is a large number of designs of viscometer in existence and although each is intended for some specific purpose it might seem at first sight unnecessary to design yet another. However the material under test - a reacting resin system - imposes constraints on the device which do not normally need to be considered. Firstly, the reactive system is being subjected to an increasing temperature. The reaction rate is exponentially related to absolute temperature and the reaction is exothermic. This means that control of the temperature can easily be lost, a condition known as exothermal runaway. The simplest, if not the only, way to avoid this state is to keep the resin mass small and to arrange for it to have a large surface to volume ratio. Secondly, on completion of the test the resin will have gelled; it will not be fully cured, but in a state such that its container and anything in it will be bonded together and must therefore be expendable. Several designs were considered including an unusual oscillatory viscometer³ but the choice ultimately fell on a simple rotating cylinder design in which the rotating part, hereafter called the probe, is a simple glass and metal rod and the resin container an ordinary test tube. A drive motor is required to rotate the probe and since a decision was made to keep the shear rate constant it was necessary to ensure a constant rotational speed. This unfortunately limited the range of viscosities which could be covered and it was found necessary to use two motors, one for viscosities up to 50 poise and the other for viscosities from 50 poise up to about 3000 poise.

2.2.2 The resin container and the probe

To contain the resin a standard commercial 12 mm × 100 mm rimless Pyrex test tube is used. Pyrex was chosen to avoid thermal failure; and the requirement to be rimless arose because of the method of assembly. A 12 mm test tube has a bore of 10 mm which when used with a 7 mm probe (a standard size of glass rod) causes the resin to be

disposed in an annulus 1.5 mm wide, thus meeting the constraint on the area/volume ratio. A sample volume of only about 3 cm³ is required which meets the second constraint of small sample mass. These items can be seen in Figs 2, 3 and 4.

To support both the resin container and the probe drive in the correct relationship, a pair of horizontal plates held 120 mm apart by three spacing pillars are bored concentrically and mounted on a support column (Fig 2). The hole in the lower plate is bushed to carry the test tube which is inserted from below, and thus has to be rimless. The upper plate is bushed to carry the motor which is inserted from above. The whole assembly can be slid up and down the support column so that the resin container can be raised from or lowered into the heater bath. In Fig 3 the unit is in the raised position as it is for assembly and in Fig 4 it is in the lowered (operational) position.

The resin probe, drawn in Fig 2 and shown in Figs 3 and 4, consists of a short length of standard 7 mm glass rod pointed at its lower end. The upper end is pushed into a steel carrier which has a 2 mm stem. The whole is immersed in resin to just above the shoulder. The stem has at its upper end a small hole to receive a 1 mm diameter wire link which connects it to the drive motor. When these components become encapsulated the test tube can be broken and the probe usually recovered. If, however, the probe is damaged then the glass can be broken out of the metal carrier and the carrier recovered.

2.2.3 The probe drive

Basically the probe requires some form of motor to rotate it and some method of measuring the torque required and hence the viscosity. Due to the constraints created by the constant speed and the fixed dimensions of resin container and probe, the range of viscosities which can be covered is limited by the sensitivity of the torque measuring device; in consequence two units are used. The first and less sensitive unit, shown in Fig 3 in the raised position, is a commercial dc motor-generator unit driven from a conventional constant speed circuit. A current shunt in the control circuit provides a signal proportional to motor current and therefore to motor torque. The sensitivity of this combination is limited by the fact that the motor losses are included in the output signal. It is however relatively robust and is used over the range 50 to 3000 poise. The more sensitive unit shown in Fig 4 in the lowered or operating position is a commercial Brookfield Rheolog viscometer head. The unit is self-contained, both drive motor and torque transducer being within the head, and is used over the range 1 to 50 poise.

The room temperature viscosity of many resin systems is very high making the accurate insertion and alignment of the probe virtually impossible in this state. Since the purpose of the test is to examine the response of the sample to a predetermined temperature cycle, it is inappropriate to pre-heat treat it and thus it is not possible to melt the sample and pour it into the container. Similarly the use of solvent is inadvisable and in order to introduce the sample into the container such subterfuges as freezing and pulverising, or shaping into rods which were then cut to length, were found to be necessary. Whatever the method, insertion of the probe is difficult and precision impossible. If precise location of the probe is not achieved then there will be errors

in the subsequent viscosity measurements and readings lower than 1 poise are unlikely to be attainable; there are, however, reasons for believing that resin systems having a minimum viscosity less than 1 poise are unsuitable for autoclave moulding¹ so that there is no reason to require measurements in this range for composite materials applications.

2.3 The heating system

2.3.1 Design considerations

Most commercial viscometers operate at steady temperatures only although a few are capable of varying the temperature linearly with time. However for the Simulator the bath temperature is required to follow the frequently complicated temperature profile experienced by material in an autoclave. Such a profile can have the following features. Firstly, the material is heated by forced convection from the enclosed gas and it follows that the temperature of the sample will rise exponentially and will be asymptotic to the gas temperature. Secondly, if the gas pressure is altered the exponential rate will also alter since it is dependent on pressure. Thirdly, it is a common production requirement that the final temperature be reached in more than one stage separated by one or more periods of constant temperature, conditions known as 'dwells'. A diagrammatic temperature profile incorporating these three features is given in Fig 5. The portion AB is the initial exponential heat-up corresponding to a gas temperature T_1 and BC is a dwell at temperature T_1 . CE is the final exponential heat-up corresponding to the gas temperature T_2 , interrupted by a change of pressure, and therefore of heating rate, at D. Thus the heating system must be able to generate exponential rates which can be altered at intermediate levels and also have the ability to maintain a constant intermediate temperature. This is achieved by an electronic network referred to as a function generator.

2.3.2 The heater bath

The heater bath, made of glass tubing in the form of a letter H, can be seen in Figs 3 and 4 and is shown schematically in Fig 6. The limbs which are about 25 mm in diameter are closed at the bottom and each has a small outlet nipple about 10 mm above the bottom which permits the limbs to be joined to a small pump. A coil of wire in one limb acts as a heater, the resin container being supported in the other. The complete bath is carried by an arm from the support column so that the appropriate limb is concentric with the resin container.

In use, oil is driven by the pump up over the heater, through the cross bar, down past the resin container and then returned to the pump. This circulation of hot oil ensures that the whole resin container is at the same temperature. Two thermocouples are immersed in the oil adjacent to the resin container and at half its depth. One supplies a feedback signal to the control system and the other is used to record the temperature on a chart.

Because both resin container and oil bath are made of glass, it is possible, (by selecting a transparent oil), to observe the behaviour of the resin during a test. For example if a resin contains solvent it is possible to note the time or temperature at

which the solvent is evolved. Because of the great affinity of resins for solvents, this temperature can be very different from the boiling temperature of the solvent; indeed it is possible by this means to note that some solvents are never evolved.

2.3.3 The temperature control

The heating control unit performs two separate functions although they are combined into one integrated whole; (a) control of the oil bath temperature and (b) generation of the required temperature profile.

The control of the bath temperature is done in a quite conventional manner. As shown in Fig 7a, a feedback signal V_i from a thermocouple T_c in the oil is compared with a demand signal V_D in a differential amplifier A to give an error signal. This error signal is fed into a power amplifier B which adjusts the power to the heater C.

The demand signal V_D is derived from a unit referred to as a function generator. In Fig 7b a clock pulse is fed into a counter E whose output feeds a digital-to-analogue converter F to give a linear ramp whose slope is a function of the counting rate. This ramp is fed into a network G which, by means of a sequence of biased diodes, reduces the slope of the ramps at each break point to give a set of six straight ramps approximating an exponential curve. In practice the linearity of the sections is not noticeable. The output of this network is the demand signal to the control unit. By adjusting the magnitude of the demand signal the maximum temperature can be controlled. The unit thus provides an output which is of exponential form but which can be varied both in the time domain (*ie* temperature rate) and in amplitude (*ie* peak temperature). The method of setting the controls is given in the Appendix.

2.4 Auxiliaries

The oil pump is driven by a 12 V dc motor and the oil heater requires a 30 V 6 A dc supply. Both of these supplies are taken from 230 V ac transformers and bridge rectifiers mounted in a case on the main frame together with the appropriate fuses, switches and indicators.

The Brookfield unit requires a supply of compressed air to energise the torque transducer; this comes from an external 240 V ac motor-driven pump which, together with a pressure transducer and safety cut-out, is mounted on a separate chassis.

The output of the torque transducer and the oil bath thermocouple are recorded on potentiometric chart recorders.

3 AN EXAMPLE OF THE USE OF THE SIMULATOR

With purpose-built devices such as this, the primary interest is in the understanding it brings to the subject rather than in a measure of its accuracy or repeatability, and the effect on the resin of a complicated cure cycle incorporating a 'dwell' will be used as an example. A 'dwell' is considered to be a simple variation of a cure cycle often used for no other purpose than to allow temperatures to equalise throughout a laminate. Furthermore its effect is commonly assumed to be minimal because it takes place at a temperature much lower than the cure temperature.

In Fig 8 the temperature curve OT is typical of the response of a composite sample to a steady gas temperature of 130°C and PQ is the corresponding viscosity curve. The definition of gelation is somewhat arbitrary but for practical purposes it may be taken to correspond to that condition after which the resin cannot be worked mechanically. For the sake of the discussion gelation is considered to have occurred at 75 poise although it can be seen that the precise value selected is immaterial since the viscosity is increasing extremely rapidly by then. OA, OB, OC, OD represent a family of simulated temperature response curves generated by increasing the gas temperature by the same amount but with the increase commencing at times a' , b' , c' and d' respectively. For each temperature curve there is a corresponding viscosity curve PA', PB', PC' or PD'. The first point to be noted is that whilst increasing the heat-up at a' advances gelation relative to the basic curve by a time A'Q' an identical temperature increase applied at b' or c' retards gelation by times Q'B' or Q'C'. Application of the temperature at d' is completely ineffective since gelation has already occurred at time Q'. These delays in application of increased temperature ab, ac and ad may be regarded as dwell times. Now consider the respective values of minimum viscosity. For both PA' and PB' the minimum is less than that of the standard PQ, and that for PC' is very similar to PQ. At time d the viscosity is so high and increasing so rapidly that an application of increased temperature is virtually ineffective.

This example of the use of the simulator shows that the effect of applying a dwell is by no means simple, but has a profound effect on the behaviour of the resin. This effect, which could not have been determined in any other way, is not what would have been expected intuitively and is markedly non-linear; this demonstrates how apparently simple operations, when examined, may prove to cause unexpectedly complex reactions.

4 CALIBRATION

Since the relationship between resin viscosity and the electrical output of the torque transducer is roughly linear, for comparative purposes it is sufficient to express the output signal of the torque transducer in electrical terms, *ie* millivolts. However, in instances in which it is desirable to have an absolute knowledge of the resin viscosity, calibration of the simulator is necessary. This can be done in principle merely by using an oil of known viscosity and measuring the torque, but since the simulator is a dynamic instrument a dynamic calibration is preferable. The viscosities of standard oils are specified at certain temperatures, which in the case of those used is 25°C . Thus, in practice, the heater bath is pre-cooled to a temperature much lower than 25°C (say 5°C) and the instrument prepared in the normal way, but filled with the standard oil in lieu of resin, and heated at a typical rate. This gives a torque/temperature relationship which can be converted to torque/viscosity at the appropriate temperature.

There is unfortunately a thermal lag through the resin container which is dependent on heat-up rate. Hence the calibration constant of the simulator is also dependent on heat-up rate. With the particular combination of resin container and probe, and for the highest rate used, the simulator has a calibration value of 4.6 poise per millivolt,

whilst for slow heat-up rates it approaches the isothermal value of 5.1 poise per millivolt. The appropriate calibration value must be employed as necessary throughout a cycle.

The use of standard commercial components is in itself a disadvantage because of manufacturing variability; 12 mm test tubes for example can have an internal diameter that varies between 9.9 mm and 10.1 mm. The torque from the simulator according to simple theory is proportional to $(R_c - R_p)/R_p^2 R_c$ where the suffices c and p refer to the container and probe respectively, R being the radius. If the same probe is always used then the variation in the test tube radius could give a variation of about 4% in the output. It is of course not difficult to discard the tubes having the highest variability and by this means errors due to this cause can be brought below any desired level without great expense. Alternatively it is possible to derive a calibration curve for the instrument such as Fig 9, which makes allowance for radius variations.

5 REPEATABILITY

There is difficulty in achieving a consistent start: the instrument cannot rotate resins of high viscosity at room temperature and therefore the start of rotation has to be delayed until the resin temperature is somewhat higher than room temperature. The heat transfer characteristics of the resin are different in the rotating and the non-rotating cases so the rate of heating of the resin and thus the initial portion of the viscosity curve depend on just when the motor is switched on. Nevertheless, repeat runs on the same batch of material suggest a repetition accuracy of about 2%, which seems adequate for practical process control applications.

6 CONCLUSIONS

The example given in section 3 shows that the simulator provides a simple but very effective means of monitoring the viscous behaviour of a reacting resin system under a particular family of predetermined temperature cycles.

A problem, which is fortunately not critical, arises from the thermal delay in the transfer of heat from the oil bath to the resin. The thermal inertia of the resin container and the resin results in the fact that the viscosity recorded when a specific bath temperature is reached is dependent on the heating rate. In practice this effect is not important since isothermal conditions are approached at those temperatures at which the viscosity is a minimum, and this is the region of greatest practical importance. In any case compensation for it can be made if necessary.

The instrument has already proved to be very useful and, as was typified in the example, has greatly added to the understanding of autoclave cure cycles and aided the evolution of practical production processes. Further, by using fast exponentials as ramps, it may also be used for similar studies of resin cure cycles associated with ovens and presses.

Acknowledgment

The author wishes to acknowledge the considerable assistance of Mr R.A. Hobbs who designed the electronic circuits.

Appendix

The operation of the function generator is controlled by six toggle switches, two rotary switches and two rotary potentiometers mounted on the face of the unit. A photograph of the assembly is given as Fig 10. The function of the various items is as follows:

A.1 NORMAL-EXTEND

This is a two-position rotary switch the operation of which is explained below.

A.2 SET POINT

This is a rotary potentiometer graduated from 100 to 200. The setting of this determines, in degrees Celsius, the steady state temperature which the heater will attain after 255 time increments of a magnitude set by the RATE switches. In the 'normal' setting of the NORMAL-EXTEND switch the temperatures are as indicated. In the 'extend' position the scale is diminished by 100 (i.e. it effectively reads from 0 to 100). In the photograph this dial is set at 177.

A.3 CHANGE POINT

This is a rotary potentiometer also graduated from 100 to 200. In the 'normal' setting of the NORMAL-EXTEND switch this control is inoperative. When it is intended to simulate a two component curve the changeover temperature is set on this dial. This represents a new (offset) zero for the second stage. It does not become operative until the selected temperature is reached at which time the NORMAL-EXTEND switch *must* be set manually to the 'extend' position. The temperature control then reverts to a new set point which is the sum of the change point setting and the set point setting diminished by 100, i.e. (change point + (set point - 100)). In the photograph the change point dial is set to 155.

A.4 NORMAL-LOW

It is sometimes necessary to simulate cycles whose end point is less than the minimum setting of the set point dial, i.e. 100°C. This is achieved in effect by using the 'change point' system with a change point value of zero. Since there is no zero position on the change point dial the operation is effected by a toggle switch. In its 'normal' position the above mentioned three controls are operative as described. In the 'low' position and with the NORMAL-EXTEND switch in 'extend' the system operates as if the change point were set to zero. In other words the set point scale is diminished by 100. The CHANGE POINT dial is inoperative.

A.5 RATE

The SET POINT temperature is reached in 255 steps where the length, in seconds, of a step is adjustable. The adjustment to the step length is made by independently setting the tens and units. The tens are set by a rotary switch that covers the range 10 to 60, whilst the units are coded in binary digits by four toggle switches (at least one of which must be on). A doubling of the range is obtained by a two position toggle switch marked N and X2. The range is thus from $(10 + 1)X1 = 11$ seconds to $(60 + 15)X2 = 105$ seconds per step. In the illustration the rate is set to 62 that is $(20 + 8 + 2 + 1)X2$.

If at any time *all* the digit switches are off then the control maintains the temperature then existing until such time as some digits are re-applied when the temperature will increase at whatever rate is then set. It is thus possible to simulate the 'dwell' condition popular with some resin manufacturers.

A.6 Accessories

There are in addition a 'start' button to initiate the cycle, a green light which pulsates at the set rate to indicate that the unit is functioning and a red light which lights when the set point is reached, *i.e.* after 255 increments.

A.7 Example

As an example of the derivation of the various rates, the case will be taken of a cycle with a change of rate and temperature typical of an autoclave cycle when pressurisation is used. In Fig 11 the full line is the temperature-time curve. The extrapolation of the first component forwards to a psuedo-asymptote and backwards to 0°C is shown dashed. The various intervals are then:

t_0	extrapolated time to 0°C	-10 min
t_1	the time to the first psuedo-asymptote	140 min
t_2	the time to the second psuedo-asymptote	170 min
t_3	the time to the changeover point	55 min
T_1	} temperature corresponding to t_1, t_2, t_3	125°C
T_2		170°C
T_3		105°C

For the initial curve the rate is $\left(\frac{140 + 10}{255}\right) 60 = 35$ seconds/step

For the second curve $\left(\frac{170 - 55}{255}\right) 60 = 27$ seconds/step

The set points are 1st = 125

2nd = $(170 - 105) + 100 = 165^\circ\text{C}$ (This will be in the extend range and will be diminished by 100.)

The change point will be 105.

With the controls set the function generator will, as soon as the start button is pressed, generate an exponential curve starting at 0°C, passing through T_3 and ending at T_1 . However at T_3 the controls are reset and a new curve is generated ending at T_2 .

An exactly similar procedure is followed for a cure cycle with dwell such as is shown in Fig 12 except that there is no change point. When t_3 is reached the four digit rate switches are set to 'off'. At the end of the dwell period they are reset to the rate required.

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Fig 1

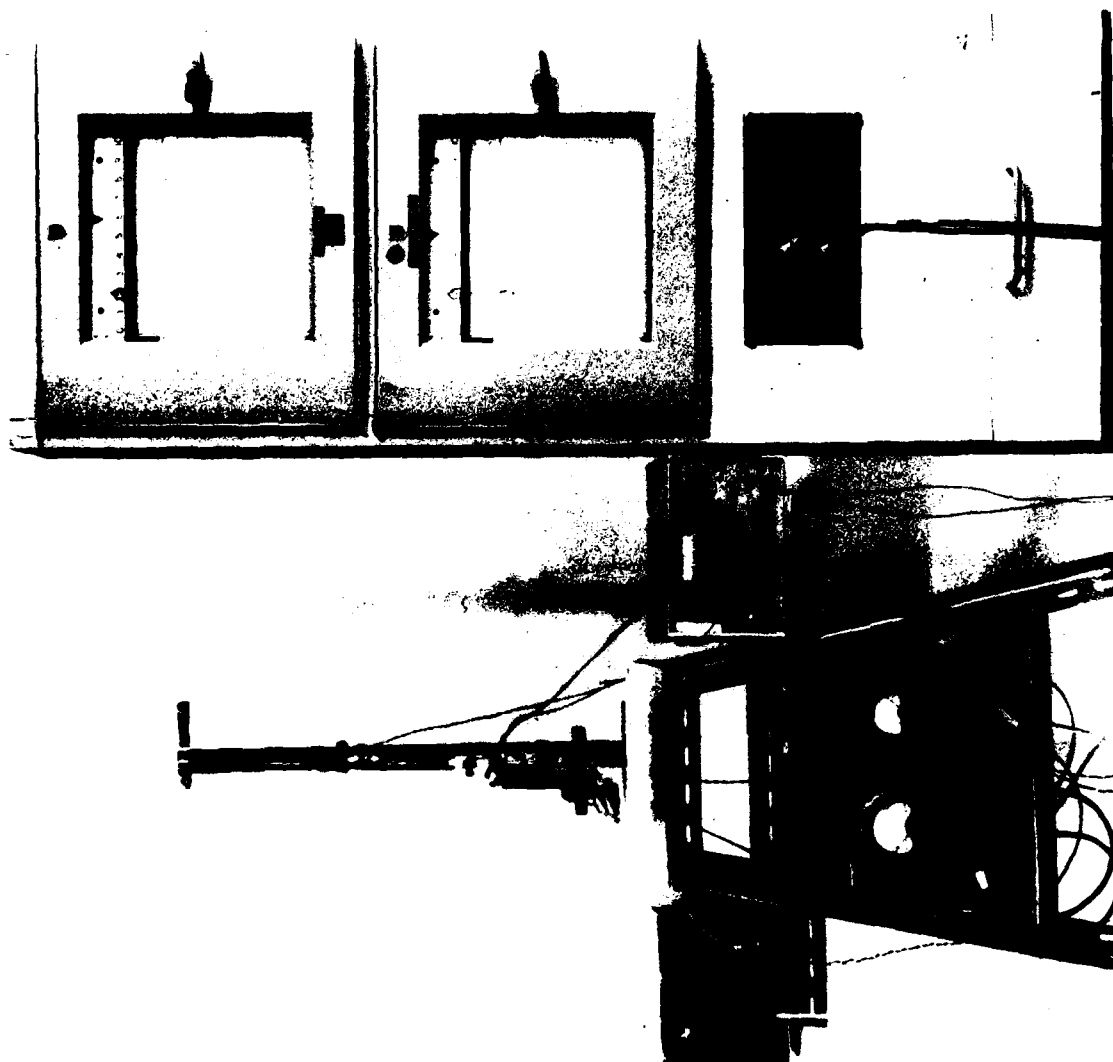


Fig 1 The complete instrument

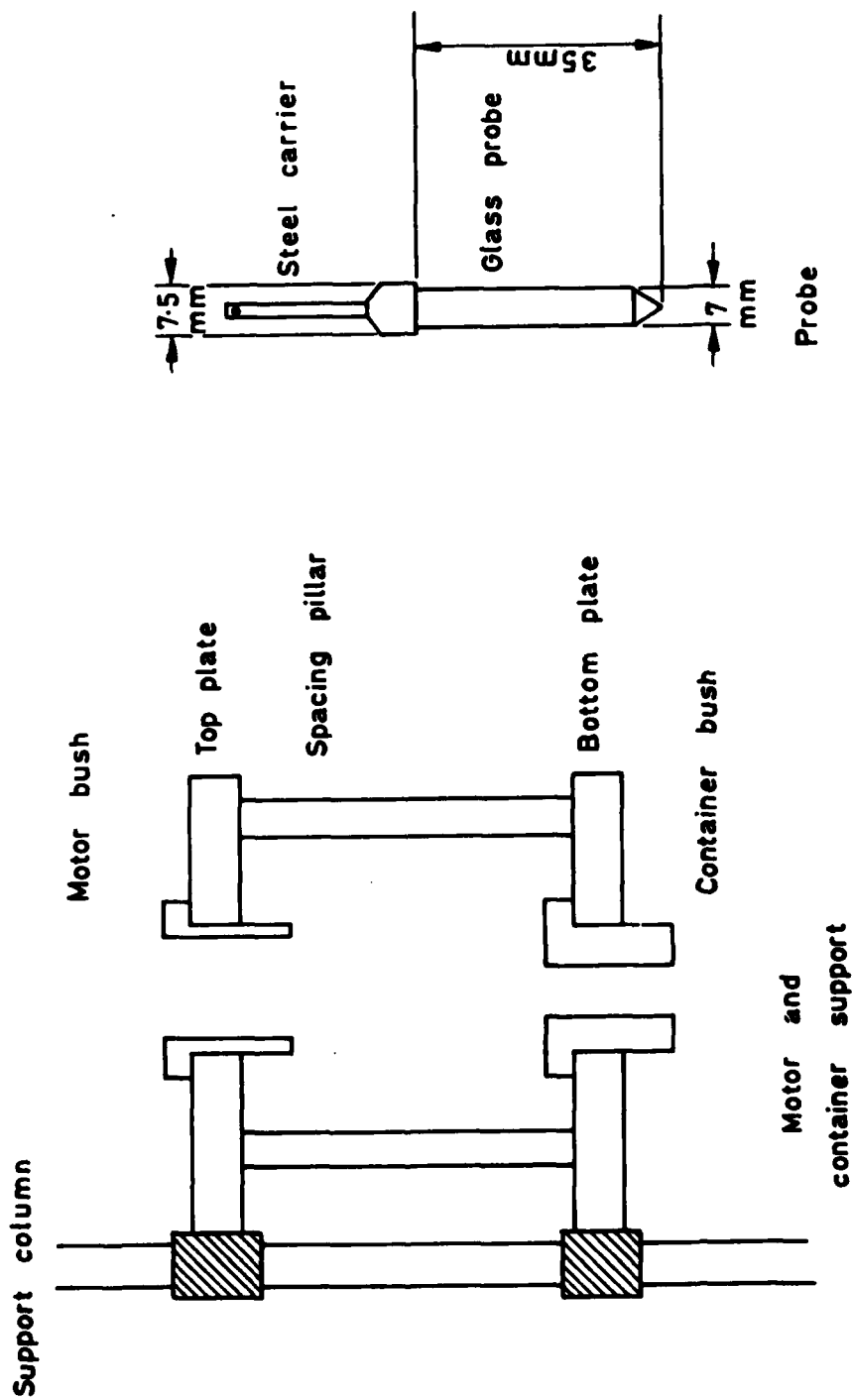


Fig 2 The support frame and the probe (diagrammatic)

Fig 3

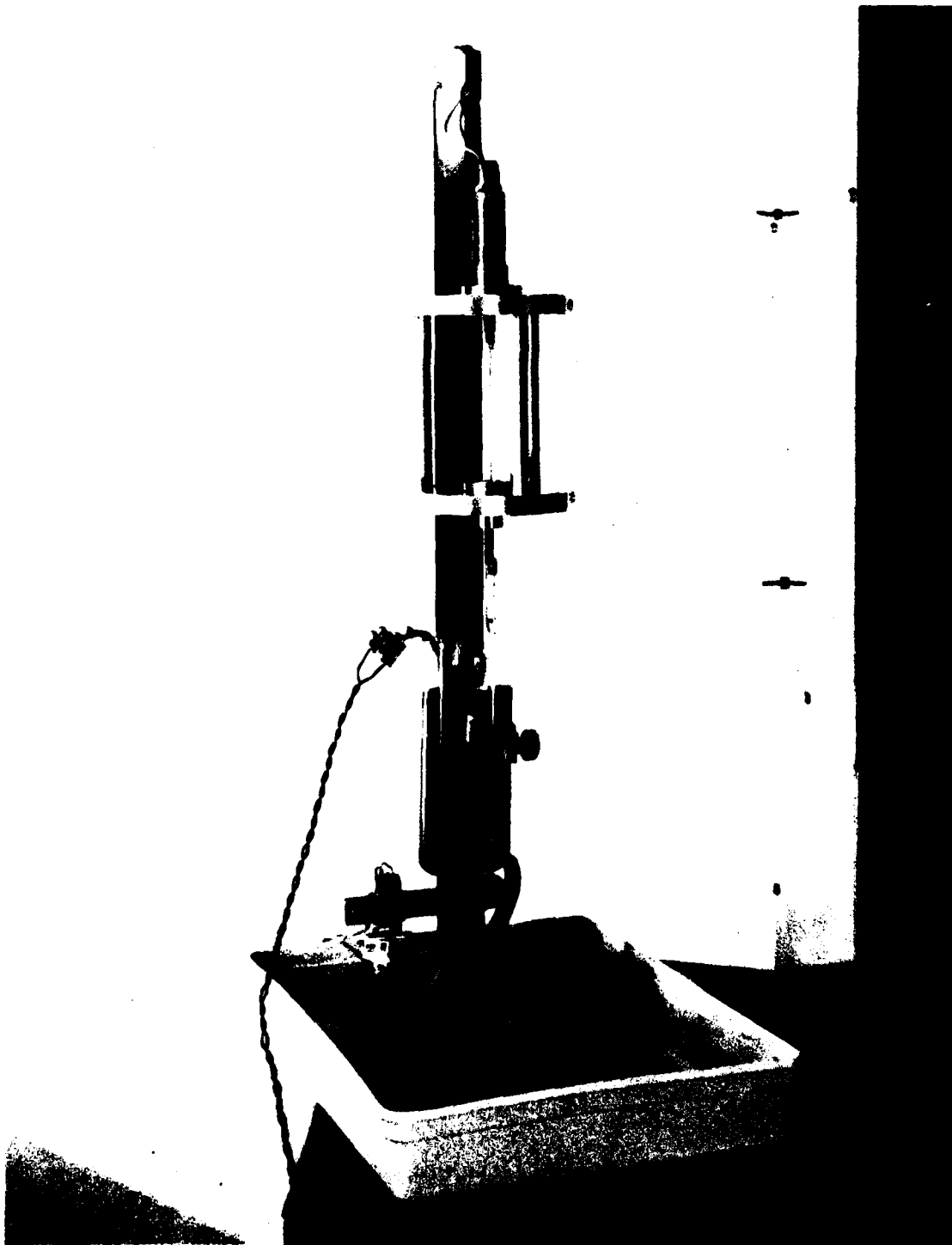


Fig 3 The model of lower sensitivity in the raised position

Fig 4

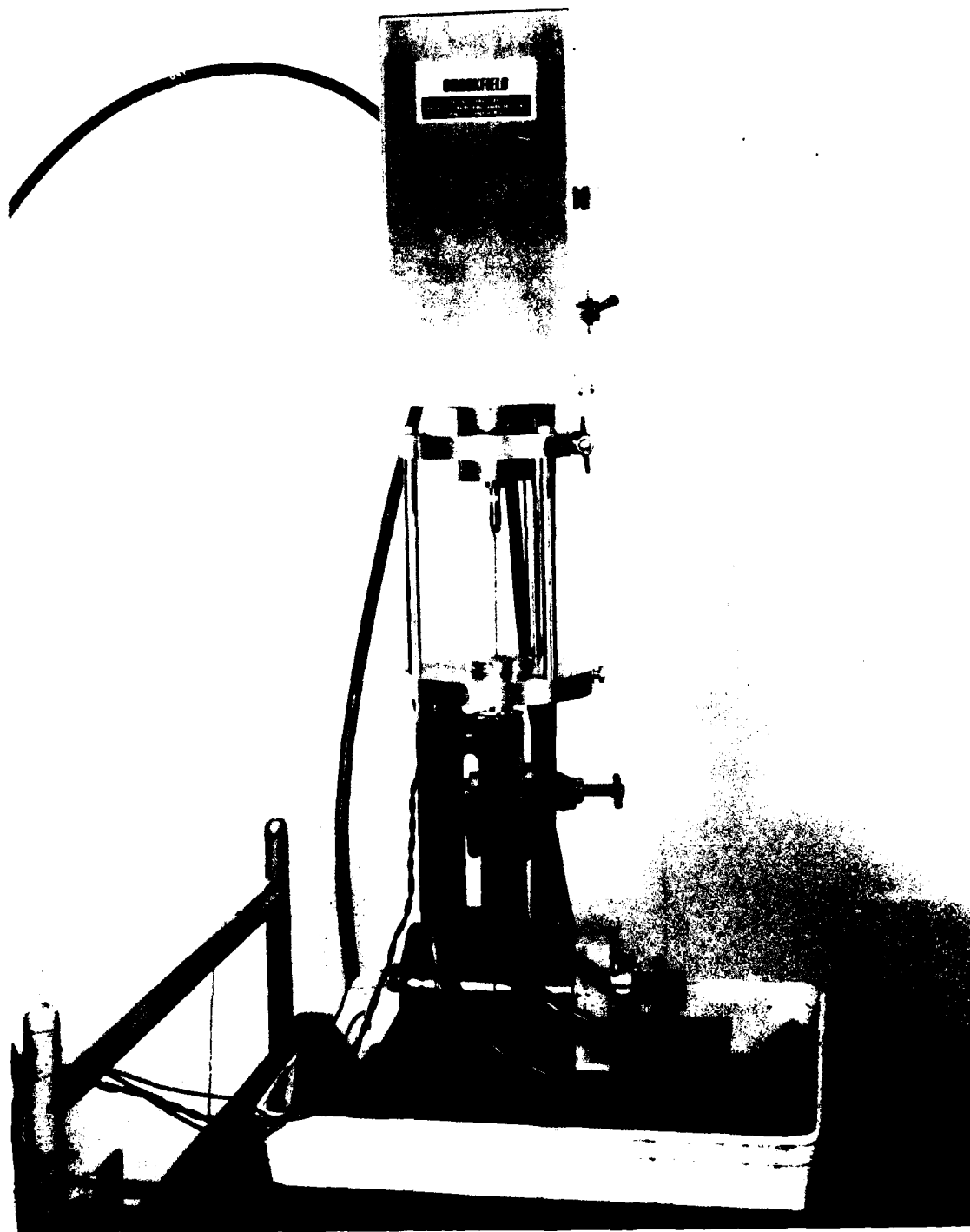


Fig 4 The model of higher sensitivity in the operating position

Figs 5&6

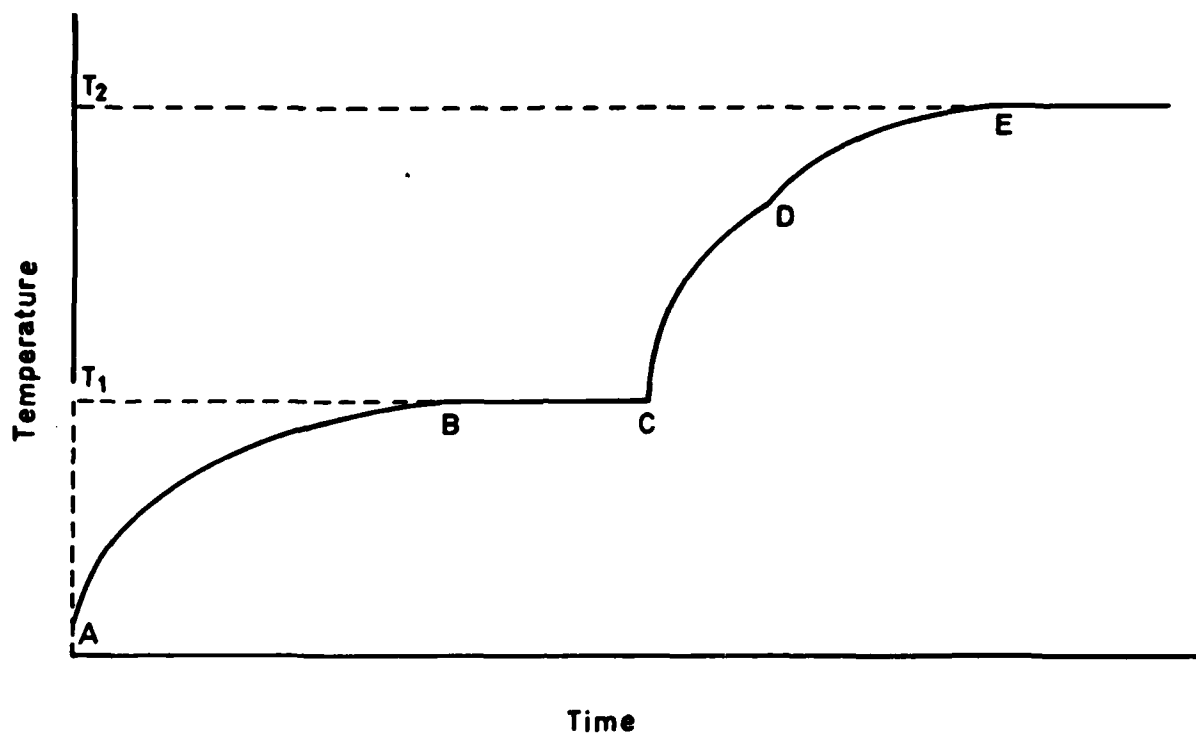


Fig 5 A typical autoclave temperature cycle

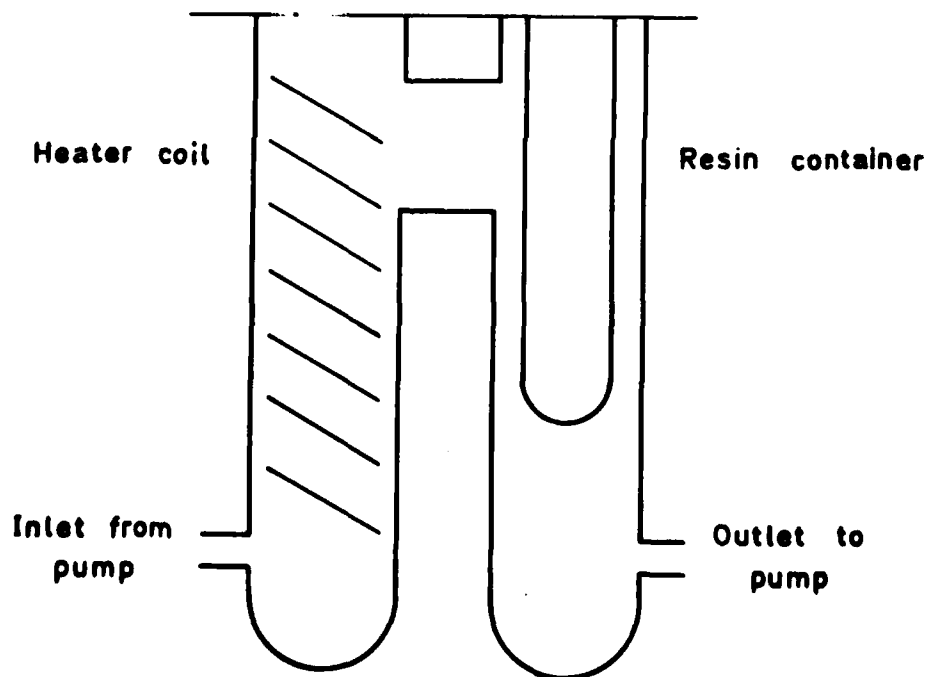
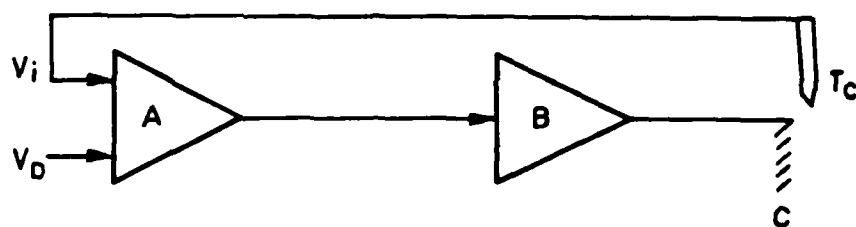
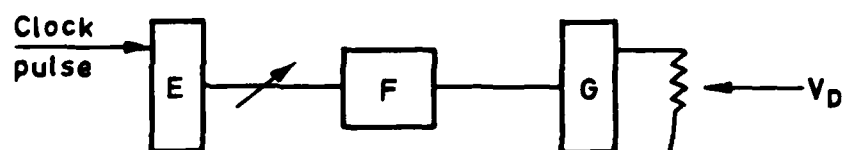


Fig 6 Diagrammatic arrangement of heater assembly



a Temperature control



b Function generator

Fig 7a&b The principle of the heating control (schematic)

Fig 8

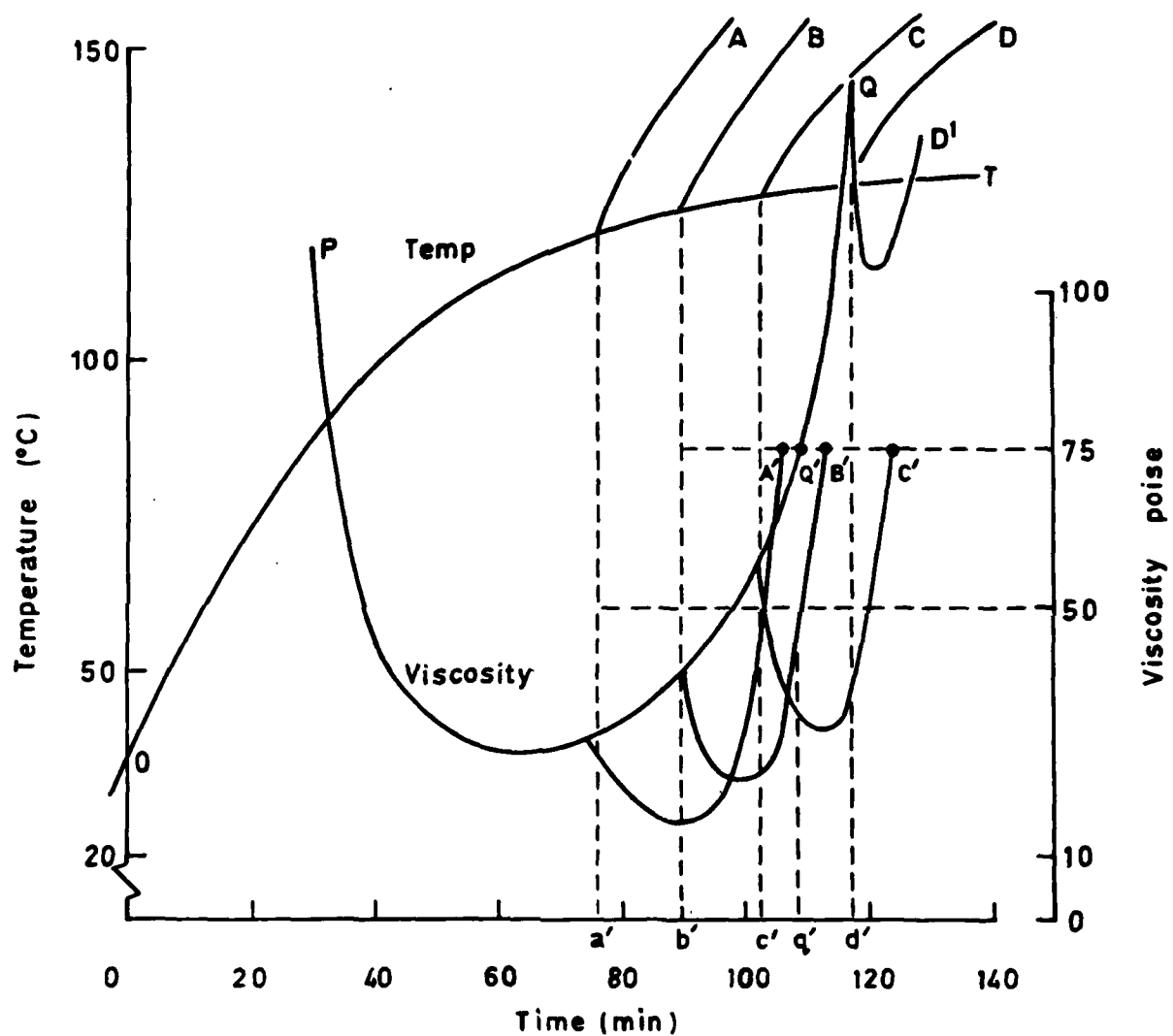


Fig 8 Viscosity curves for a family of temperature profiles

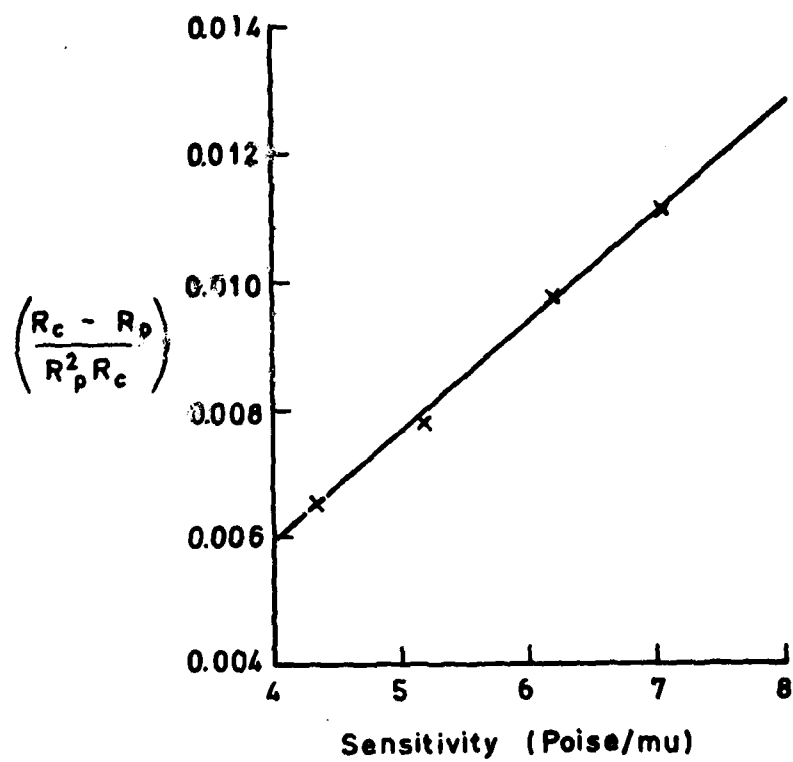
Fig 9 Variation of sensitivity with container radius (R_c)

Fig 10

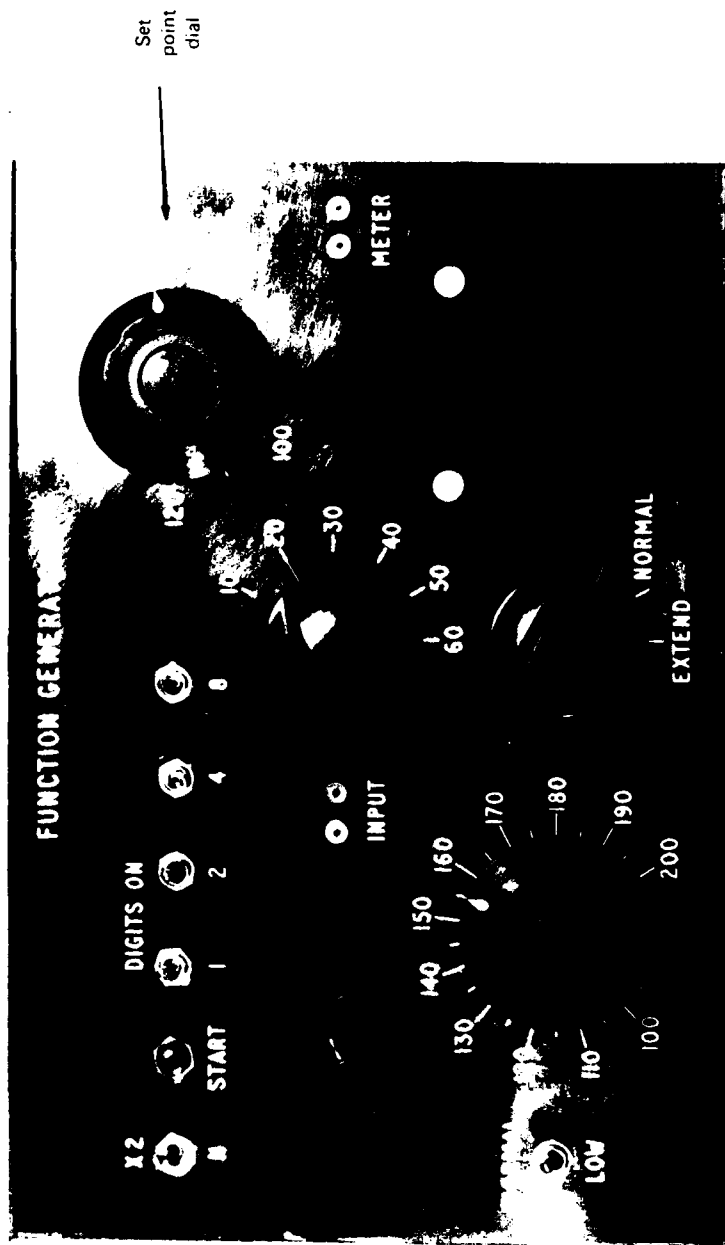


Fig 10 The heating control unit

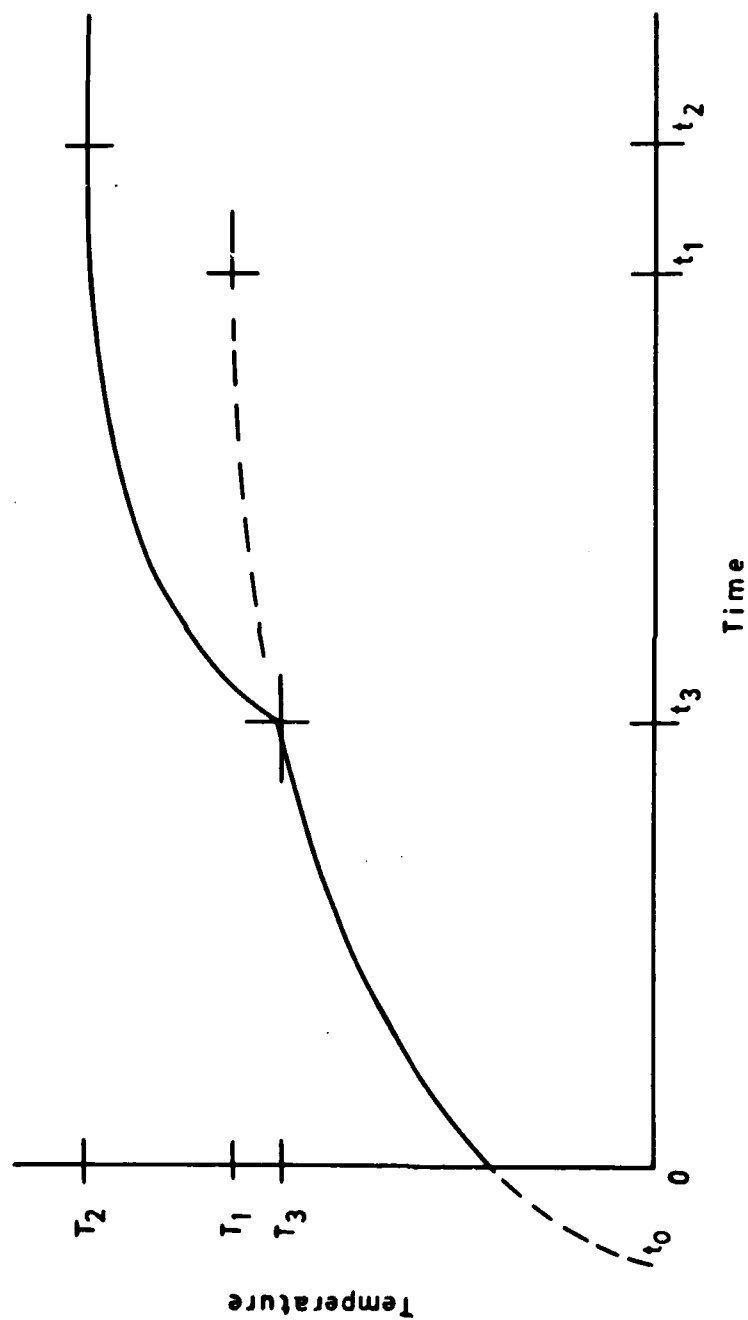


Fig 11 Derivation of settings for a typical autoclave temperature cycle

Fig 12

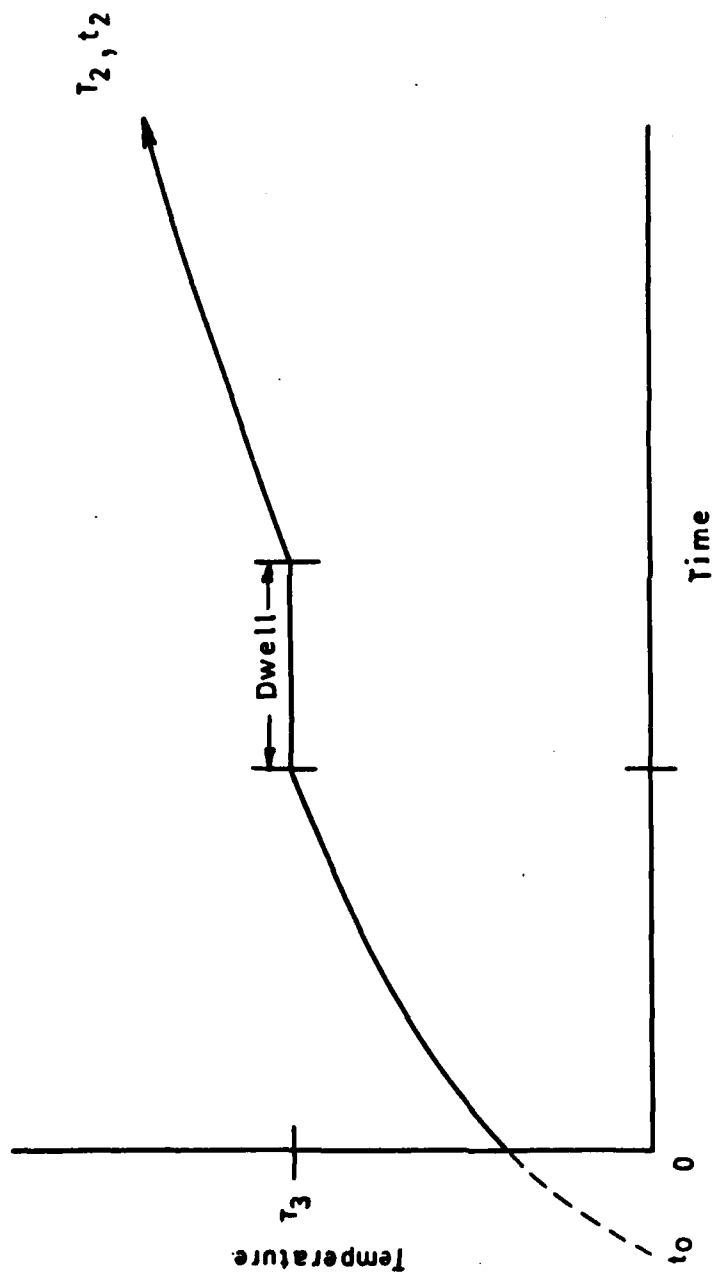


Fig 12 Derivation of settings for a temperature cycle with dwell

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1. DRIC Reference (to be added by DRIC)	2. Originator's Reference RAE TR 81145	3. Agency Reference N/A	4. Report Security Classification/Marking		
5. DRIC Code for Originator 7673000W		6. Originator (Corporate Author) Name and Location Royal Aircraft Establishment, Farnborough, Hants, UK			
5a. Sponsoring Agency's Code N/A		6a. Sponsoring Agency (Contract Authority) Name and Location N/A			
7. Title The design and use of a viscometer for the study of reacting resin systems					
7a. (For Translations) Title in Foreign Language					
7b. (For Conference Papers) Title, Place and Date of Conference					
8. Author 1. Surname, Initials Childs, R.	9a. Author 2	9b. Authors 3, 4	10. Date November 1981	Pages 23	Refs. 3
11. Contract Number N/A	12. Period N/A	13. Project	14. Other Reference Nos. Structures BF/B/0898		
15. Distribution statement (a) Controlled by - (b) Special limitations (if any) -					
16. Descriptors (Keywords) (Descriptors marked * are selected from TEST) Viscometry. Resin cure cycles. Composite materials.					
17. Abstract This Report describes a viscometry system which has been developed specifically to study reacting resin systems under predetermined temperature cycles such as those representative of the cure conditions used for carbon fibre reinforced epoxy resin laminates. The constraints placed upon the design both by the need to reduce the number of variables and by the nature of the material under test are discussed together with the methods adopted to overcome them. By use of an example, the viscometry system described is shown to provide a detailed understanding of what, at first sight, appears to be a simple process but is in fact unexpectedly complex.					